identity, crystallinity, residue on ignition, and absorptivity.

(*b*) The batch for potency and moisture.

(ii) Samples required:

(*a*) The minocycline hydrochloride used in making the batch: 10 packages, each containing approximately 300 milligrams.

(*b*) The batch: A minimum of 30 capsules.

(b) *Tests and methods of assay*—(1) *Potency*. Proceed as directed in § 446.60(b)(1) of this part, except prepare the sample solution and calculate the minocycline potency as follows:

(i) *Sample solution*. Open a representative number of capsules and empty the contents into a volumetric flask containing mobile phase (described in § 446.60(b)(1)(i)(*c*) of this part) and shake to dissolve. Dilute with mobile phase to give a stock solution of convenient concentration. Filter the stock solution. Remove an aliquot of the stock solution and further dilute with mobile phase to obtain a solution containing 500 micrograms of minocycline activity per milliliter (estimated). Use this solution within 3 hours of preparation.

(ii) *Calculations.* Calculate the minocycline content as follows:

$$\text{Milligrams of minocycline per capsule} = \frac{A_u \times P_s \times d}{A_s \times 1{,}000 \times n}$$

where:

A_u= Area of the minocycline peak in the chromatogram of the sample (at a retention time equal to that observed for the standard);

A_s= Area of the minocycline peak in the chromatogram of the minocycline working standard:

P_s= Minocycline activity in the minocycline working standard solution in micrograms per milliliter;

d = Dilution factor of the sample; and

n = Number of capsules in the sample assayed.

(2) *Moisture.* Proceed as directed in § 436.201 of this chapter.

[39 FR 19076, May 30, 1974, as amended at 43 FR 11163, Mar. 17, 1978; 44 FR 22058, Apr. 13, 1979; 50 FR 19920, May 13, 1985; 53 FR 32609, Aug. 26, 1988]

§ 446.160c Minocycline hydrochloride oral suspension.

(a) *Requirements for certification*—(1) *Standards of identity, strength, quality, and purity.* Minocycline hydrochloride oral suspension is minocycline hydrochloride with one or more suitable flavorings, wetting agents, preservatives, and diluents in an aqueous vehicle. Each milliliter contains minocycline hydrochloride equivalent to 10 milligrams of minocycline. Its potency is satisfactory if it is not less than 90 percent and not more than 130 percent of the number of milligrams of minocycline that it is represented to contain. Its pH is not less than 7.0 and not more than 9.0. The minocycline hydrochloride used conforms to the standards prescribed by § 446.60(a)(1).

(2) *Labeling.* It shall be labeled in accordance with the requirements of § 432.5 of this subchapter.

(3) *Requests for certification; samples.* In addition to complying with the requirements of § 431.1 of this chapter, each such request shall contain:

(i) Results of tests and assays on:

(*a*) The minocycline hydrochloride used in making the batch for potency, moisture, pH, epi-minocycline content, identity, crystallinity, residue on ignition, and absorptivity.

(*b*) The batch for potency and pH.

(ii) Samples required:

(*a*) The minocycline hydrochloride used in making the batch: 10 packages, each containing approximately 300 milligrams.

(*b*) The batch: A minimum of five immediate containers.

(b) *Tests and methods of assay*—(1) *Potency*. Proceed as directed in § 446.60(b)(1) of this part, except prepare the sample solution and calculate the minocycline potency as follows:

(i) *Sample solution*. Transfer an accurately measured 5-milliliter portion of the well-shaken suspension to a 100-milliliter volumetric flask. Dilute to mark with mobile phase (described in § 446.60(b)(1)(i)(*c*) of this part) and mix well. Filter this solution and use within 3 hours of its preparation.

(ii) *Calculations.* Calculate the minocycline content as follows:

$$\text{Milligrams of minocycline per milliliter} = \frac{A_u \times P_s \times d}{A_s \times 1{,}000 \times 5}$$

where:

A_u = Area of the minocycline peak in the chromatogram of the sample (at a retention time equal to that observed for the standard);

A_s = Area of the minocycline peak in the chromatogram of the minocycline working standard:

P_s = Minocycline activity in the minocycline working standard solution in micrograms per milliliter; and

d = Dilution factor of the sample.

(2) *pH.* Proceed as directed in §436.202 of this subchapter, using the undiluted sample.

[39 FR 19076, May 30, 1974, as amended at 43 FR 11163, Mar. 17, 1978; 44 FR 22058, Apr. 13, 1979; 50 FR 19920, May 13, 1985; 53 FR 32609, Aug. 26, 1988]

§446.165 Oxytetracycline oral dosage forms.

§446.165a Oxytetracycline tablets.

(a) *Requirements for certification*—(1) *Standards of identity, strength, quality, and purity.* Oxytetracycline tablets are tablets composed of oxytetracycline and one or more suitable and harmless, diluents, binders, lubricants, colorings, and coating substances. The potency of each tablet is 250 milligrams of oxytetracycline. Its potency is satisfactory if it is not less than 90 percent and not more than 120 percent of the number of milligrams of oxytetracycline that it is represented to contain. The moisture content is not more than 7.5 percent. They shall disintegrate within 1 hour. The oxytetracycline used conforms to the standards prescribed by §446.65(a)(1).

(2) *Labeling.* It shall be labeled in accordance with the requirements of §432.5 of this chapter.

(3) *Requests for certification; samples.* In addition to complying with the requirements of §431.1 of this chapter, each such request shall contain:

(i) Results of tests and assays on:

(*a*) The oxytetracycline used in making the batch for potency, moisture, pH, absorptivity, identity, and crystallinity.

(*b*) The batch for potency, moisture, and disintegration time.

(ii) Samples required:

(*a*) The oxytetracycline used in making the batch: 10 packages, each containing approximately 300 milligrams.

(*b*) The batch: A minimum of 36 tablets.

(b) *Tests and methods of assay*—(1) *Potency.* Proceed as directed in §436.106 of this chapter, preparing the sample for assay as follows: Place a representative number of tablets into a high-speed glass blender jar containing sufficient 0.1*N* hydrochloric acid to obtain a stock solution of convenient concentration containing not less than 150 micrograms of oxytetracycline per milliliter (estimated). Blend for 3 to 5 minutes. Remove an aliquot of the stock solution and further dilute with sterile distilled water to the reference concentration of 0.24 microgram of oxytetracycline per milliliter (estimated).

(2) *Moisture.* Proceed as directed in §436.201 of this chapter.

(3) *Disintegration time.* Proceed as directed in §436.212 of this chapter, using the method described in paragraph (e)(1) of that section.

[43 FR 11163, Mar. 17, 1978; 43 FR 34456, Aug. 4, 1978, as amended at 50 FR 19920, May 13, 1985]

§§446.165b—446.165c [Reserved]

§446.165d Oxytetracycline for oral suspension.

(a) *Requirements for certification*—(1) *Standards of identity, strength, quality, and purity.* Oxytetracycline for oral suspension is oxytetracycline with one or more suitable and harmless buffer substances, preservatives, diluents, colorings, and flavorings. When prepared as directed in the labeling, each milliliter contains 50 milligrams of oxytetracycline. Its potency is satisfactory if it is not less than 90 percent and not more than 115 percent of the number of milligrams of oxytetracycline that it is represented to contain. Its loss on drying is not more than 2 percent. When reconstituted as directed in the labeling, its pH is not less than 5.5 and not more than 7.5. The oxytetracycline used conforms to the standards prescribed by §446.65(a)(1).

(2) *Labeling.* It shall be labeled in accordance with the requirements of §432.5 of this chapter.